

EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	129	membrane and pore size and nanoparticles same gold	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:01
L2	3	1 and leach	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:02
L3	2	1 and leaching	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:01
L4	2	2 and iodine	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:01
L5	2	3 and iodine	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:06
L6	2610	membrane and gold and iodine	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:06
L7	8	6 and pores and particles and extracting same particles	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:07
L8	72	("2947646" "3082109" "3351495" "3514607" "3745142" "3870593" "4247498" "4324811" "4485838" "4539256" "4550123" "4562108" "4619963" "4650730" "4726989").PN. OR ("4957943").URPN.	US-PGPUB; USPAT; USOCR	AND	ON	2006/08/11 15:28
L9	72	("2947646" "3082109" "3351495" "3514607" "3745142" "3870593" "4247498" "4324811" "4485838" "4539256" "4550123" "4562108" "4619963" "4650730" "4726989").PN. OR ("4957943").URPN.	US-PGPUB; USPAT; USOCR	AND	ON	2006/08/11 15:35
L10	0	membrane and microporosus and particles and gold and iodine	USPAT; EPO; DERWENT	AND	ON	2006/08/11 15:37
L11	58	("3231530" "3471597" "3679528" "3719640" "3801692" "3843761" "3853601" "3880966" "3951815" "4039440" "4051300" "4238459" "4340482" "4346142" "4432875" "4900449" "5013439" "5085775" "5098569" "5294346").PN.	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:26
L12	0	"4957943.PN"	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:26

EAST Search History

L13	61	"4957943"	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:39
L14	2	13 AND GOLD AND IODINE	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:42
L15	1	making same microporous membrane and particles same gold	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:43
L16	0	15 and gold adj4 iodine	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:43
L17	40	gold adj5 iodine and membrane	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:44
L18	1	17 and membrane adj5 pores	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:44
L19	15	17 porous	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:49
L20	24	"4557759"	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:53
L21	654	membrane and leaching gold	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:53
L22	73	21 and iodine	USPAT; EPO; DERWENT	AND	ON	2006/08/11 16:53

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Search Results - Record(s) 1 through 9 of 9 returned.

☐ 1. Document ID: US 5942098 A

L14: Entry 1 of 9

File: USPT

Aug 24, 1999

US-PAT-NO: 5942098

DOCUMENT-IDENTIFIER: US 5942098 A

TITLE: Method of treatment of water and method and composition for recovery of precious metal

DATE-ISSUED: August 24, 1999

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Sekissov; Artuor	Las Vegas	NV		
Paronyan; Aramais	Los Angeles	CA		
Kouzin; Vladimir	Las Vegas	NV		
Lalabekyan; Natella	Los Angeles	CA		

US-CL-CURRENT: [205/565](#), [205/687](#), [205/746](#), [205/770](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KIMC	Draw D
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☐ 2. Document ID: US 5620585 A

L14: Entry 2 of 9

File: USPT

Apr 15, 1997

US-PAT-NO: 5620585

DOCUMENT-IDENTIFIER: US 5620585 A

TITLE: Inorganic perbromide compositions and methods of use thereof

DATE-ISSUED: April 15, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Dadgar; Ahmad	West Lafayette	IN		
Howarth; Jonathan N.	West Lafayette	IN		
Sergeant; Rodney H.	West Lafayette	IN		
Favstritsky; Nicolai A.	West Lafayette	IN		
McKeown; Julie A.	West Lafayette	IN		

Borden; Dennis W.	West Lafayette	IN
Sanders; Brent M.	West Lafayette	IN
Likens; Jane	West Lafayette	IN

US-CL-CURRENT: [205/565](#); [205/566](#), [205/568](#), [205/571](#), [205/619](#), [205/625](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw D
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☐ 3. Document ID: US 5607619 A

L14: Entry 3 of 9

File: USPT

Mar 4, 1997

US-PAT-NO: 5607619

DOCUMENT-IDENTIFIER: US 5607619 A

TITLE: Inorganic perbromide compositions and methods of use thereof

DATE-ISSUED: March 4, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Dadgar; Ahmad	West Lafayette	IN		
Howarth; Jonathan N.	West Lafayette	IN		
Sergeant; Rodney H.	West Lafayette	IN		
Favstritsky; Nicolai A.	West Lafayette	IN		
McKeown; Julie A.	West Lafayette	IN		
Borden; Dennis W.	West Lafayette	IN		
Sanders; Brent M.	West Lafayette	IN		
Likens; Jane	West Lafayette	IN		

US-CL-CURRENT: [252/187.2](#); [423/22](#), [423/23](#), [423/27](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC	Draw D
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☐ 4. Document ID: US 5169503 A

L14: Entry 4 of 9

File: USPT

Dec 8, 1992

US-PAT-NO: 5169503

DOCUMENT-IDENTIFIER: US 5169503 A

TITLE: Process for extracting metal values from ores

DATE-ISSUED: December 8, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Baughman; David R.	Golden	CO	80401	
Huff; Ray V.	Golden	CO	80401	

Axen; Steven G. Golden CO 80401

US-CL-CURRENT: 205/569; 423/38, 423/40, 423/658.5

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC	Draw D
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☐ 5. Document ID: US 5051128 A

L14: Entry 5 of 9

File: USPT

Sep 24, 1991

US-PAT-NO: 5051128

DOCUMENT-IDENTIFIER: US 5051128 A

TITLE: Elution process for gold-iodine complex from ion-exchange resins

DATE-ISSUED: September 24, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kubo; Susumu	Sapporo			JP

US-CL-CURRENT: 75/712; 423/24, 423/38, 75/736, 75/737

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC	Draw D
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☐ 6. Document ID: US 5026420 A

L14: Entry 6 of 9

File: USPT

Jun 25, 1991

US-PAT-NO: 5026420

DOCUMENT-IDENTIFIER: US 5026420 A

TITLE: Purification process for gold-bearing iodine lixiviant

DATE-ISSUED: June 25, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kubo; Susumu	Minami-ku, Sapporo City, Hokkaido			JP

US-CL-CURRENT: 75/712; 423/139, 423/24, 423/49, 423/DIG.14

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMIC	Draw D
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☐ 7. Document ID: US 4734171 A

L14: Entry 7 of 9

File: USPT

Mar 29, 1988

US-PAT-NO: 4734171

DOCUMENT-IDENTIFIER: US 4734171 A

TITLE: Electrolytic process for the simultaneous deposition of gold and replenishment of elemental iodine

DATE-ISSUED: March 29, 1988

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Murphy; Jack W.	Laramie	WY		

US-CL-CURRENT: 205/566; 205/570, 423/38, 423/39, 423/42, 423/46, 423/500

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC	Draw D
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☐ 8. Document ID: US 4637865 A

L14: Entry 8 of 9

File: USPT

Jan 20, 1987

US-PAT-NO: 4637865

DOCUMENT-IDENTIFIER: US 4637865 A

**** See image for Reexamination Certificate ****

TITLE: Process for metal recovery and compositions useful therein

DATE-ISSUED: January 20, 1987

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Sergent; Rodney H.	West Lafayette	IN		
Thanstrom; Kenneth N.	Sylmar	CA		

US-CL-CURRENT: 205/569; 423/109, 423/150.1, 423/38, 423/39, 423/98

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC	Draw D
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☐ 9. Document ID: US 4557759 A

L14: Entry 9 of 9

File: USPT

Dec 10, 1985

US-PAT-NO: 4557759

DOCUMENT-IDENTIFIER: US 4557759 A

TITLE: Iodine leach for the dissolution of gold

DATE-ISSUED: December 10, 1985

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
McGrew; Kent J.	Laramie	WY		

Murphy; Jack W.

Laramie

WY

US-CL-CURRENT: [75/712](#); [205/570](#), [299/10](#), [423/38](#), [423/39](#), [423/42](#), [423/46](#)

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMID	Draw D
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Terms	Documents
L13 and iodine	9

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Freeform Search

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	JPO Abstracts Database
	Derwent World Patents Index
	IBM Technical Disclosure Bulletins

Term:	L13 and iodine	 
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Search History

DATE: Friday, August 11, 2006 [Printable Copy](#) [Create Case](#)

Set Name Query

side by side

DB=USPT; PLUR=YES; OP=ADJ

Hit Count Set Name

result set

<u>L14</u>	L13 and iodine	9	<u>L14</u>
<u>L13</u>	membrane and leaching same gold	103	<u>L13</u>
<u>L12</u>	leaching nanoparticles same membrane	0	<u>L12</u>
<u>L11</u>	l1 and choloaurate	0	<u>L11</u>
<u>L10</u>	1 and choloaurate	0	<u>L10</u>
<u>L9</u>	l1 and extracting gold	3	<u>L9</u>
<u>L8</u>	L6 and iodate	0	<u>L8</u>
<u>L7</u>	L6 and iodine	0	<u>L7</u>
<u>L6</u>	1 and choroaurate	3	<u>L6</u>
<u>L5</u>	l2 and choloaurate	0	<u>L5</u>
<u>L4</u>	2 and choloaurate	0	<u>L4</u>
<u>L3</u>	L2 and leaching	6	<u>L3</u>
<u>L2</u>	L1 and iodine	166	<u>L2</u>
<u>L1</u>	membrane and gold same particles and pores	1330	<u>L1</u>

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Generate Collection

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L16: Entry 3 of 3

File: USPT

Jan 16, 1979

DOCUMENT-IDENTIFIER: US 4134837 A

TITLE: Ethylene-vinyl alcohol copolymer membranes having improved permeability characteristics and a method for producing the same

Detailed Description Text (12):

The structure of the membrane produced as above according to the present invention was examined with a scanning electron microscope. The dry membrane was frozen with liquid nitrogen and broken so that it presents a fracture cross-section. This sectional area was coated with gold to a thickness of 100 Angstrom units and examined under an electron microscope. Using an accelerating voltage of 20 KV, the secondary electron image was observed and photographed.

Detailed Description Text (47):

Then, the ethylene-vinyl alcohol copolymer membrane was subjected to an elution test. The results are shown in Table 2, together with the corresponding data on the cuprophane membrane. The above elution test was performed in the following manner. The sample membrane was cut to 1.5 cm square, and 2 grams of the specimens were heated together with 100 ml of distilled water at 70.degree. C for predetermined time periods. Ten ml. of the extract was taken, and following the addition of 20 ml of a 0.01 N-aqueous solution of potassium permanganate and 1 ml of a 3N-aqueous solution of sulfuric acid, the extract was boiled for 3 minutes and, then, allowed to cool. Then, 1 ml of an aqueous solution of potassium iodide (10 wt. %) was added, whereupon iodine was liberated to turn the solution from violet to reddish yellow. This liberated iodine was titrated with sodium thiosulfate and the difference from the blank was taken as the amount of potassium permanganate consumed. One ml of an aqueous solution of starch (1 %) was added as an indicator. Of course, a fresh extract was used for each of the elution runs.

Current US Cross Reference Classification (6):

264/41

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L16: Entry 2 of 3

File: USPT

Aug 19, 2003

DOCUMENT-IDENTIFIER: US 6608117 B1

TITLE: Methods for the preparation of cellular hydrogels

Brief Summary Text (1):

The invention disclosed herein deals in one embodiment with cellular hydrogels and methods for their preparation. The hydrogels of this invention can be colored, rendered radio opaque, or can be complexed, for example, with iodine and/or other germicides to yield useful materials.

Brief Summary Text (31):

U.S. Pat. No. 5,071,648 teaches a composition comprising complexes between acetalized PVA and iodine, useful in antimicrobial applications such as in controlling infection by releasing a desired amount of iodine as an antimicrobial agent. The articles made using this complex are useful in a number of applications, especially in wound dressings.

Brief Summary Text (32):

In other antimicrobial applications, U.S. Pat. Nos. 5,774,150 and 5,928,665 teach methods of preparation of an antimicrobial material by creating a complex between iodine and acetalized foam. Acetalized PVA foam can also be impregnated with a polyol from water solution to create soft foam that will not dry in air nor will it become rigid. The impregnation with polyol has another advantage, that is, its presence accelerates formation of iodates and iodine oxide.

Brief Summary Text (40):

Yet another objective is to disclose methods to color and/or make such PVA hydrogels radio opaque and finally, there is an objective of disclosing the complexing of iodine and the PVA hydrogels and the binding of other germicidal agents to such PVA matrices in the cellular PVA hydrogels.

Detailed Description Text (26):

The cellular materials of this invention can be colored or rendered radio opaque. Such additives are added before or at the time that the pore forming materials are added, or at the time that the frothing step is performed in the frothing method. Such radio opaque materials are those having high electron densities and include, but are not limited to, barium sulfate, bismuth suboxide, gold and the like. Radio opaque materials can be added in various amounts to cellular PVA hydrogels, usually at the level of from 1 to about 35 weight percent based on the weight of PVA. When higher visual contrast is desired, the higher levels of radio opaque material need to be incorporated into the walls of cellular PVA hydrogels. The cellular materials of this invention are colored with non-elutable, non-toxic pigments or dyes, typically by dispersing the colorant in the PVA solution before the addition of the pore forming material. Coloring can also be done after the pore-forming materials have been removed from physically cross-linked PVALmatrices by placing the sponge into the solution containing the desired colorant. The colorant can be stabilized in cellular PVA matrix by drying and then heating the dry cellular PVA matrix which may induce chemical binding, when possible, between the dye and the PVA which would then immobilize the colorant. The cellular materials of the invention can also be plasticised and thus can be made permanently flexible by incorporating suitable plasticizers, such as, but not limited to polyhydric alcohols having 2 to 6 carbon

atoms and 2 to 3 hydroxyl groups, particularly alkane diols and triols and especially ethylene glycol, propylene glycol, diglycols, diethylene glycol, triethylene glycol, dipropylene glycol, tripropylene glycol, neopentyl glycol, triglycols, glycerin (1,2,3-propanetriol), trimethylol propane, trimethylol ethane, triethylene glycol, propanediol, butanediol, hexanediol and hexanetriol, polyethylene and polypropylene glycols of various molecular weight, trimethyl or triethyl citrate, methyl or ethyl lactate, methyl or ethyl glycolate or 2-ethyl-2 (hydroxymethyl)-1,3-propanediol and the mixtures thereof. The use of triethylene glycol is especially preferred for plastization of the sponges used in topical applications and the use of glycerin when the plasticizer needs to have low cytotoxicity.

Detailed Description Text (40):

As alluded to earlier in this specification, the hydrogels of this invention can be complexed with iodine and can be combined with other germicides. The iodine complex has an extraordinary antimicrobial activity while providing a soft and gentle sponge that does not adhere to the tissue. Furthermore, it provides a color indication of the status of its antimicrobial activity. It is well known that iodine is an outstanding microbiocide having an extraordinary range of action while having low cytotoxicity at low iodine concentrations. For example, iodine is especially desirable for use against bacteria, tubercle bacilli, fungi, protozoa and viruses. Using iodine complexes with the cellular PVA hydrogels iodine can be delivered in a controlled manner that thus will overcome the typical problem of delivery of iodine to tissue at the desired rate and time period. When cellular hydrogel forms a complex with iodine it has a golden color until used, but when applied to tissue it assumes an active black color.

Detailed Description Text (41):

When iodine is consumed the black color disappears which provides a visual indication of the activity status of iodine in the wound dressing. Use of plasticizers is also desired to permanently soften cellular PVA hydrogels complexed with iodine.

Current US Cross Reference Classification (1):

264/41

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L16: Entry 1 of 3

File: USPT

Feb 15, 2005

DOCUMENT-IDENTIFIER: US 6855743 B1

TITLE: Reinforced, laminated, impregnated, and composite-like materials as crosslinked polyvinyl alcohol hydrogel structures

Brief Summary Text (24):

Still another object of this invention is to provide methods of coloring and/or making polyvinyl alcohol hydrogel compositions and articles Radio opaque and to provide methods of complexing such polyvinyl alcohol matrices with iodine and/or other germicidal agents or disinfectants that can be fashioned into useful articles or structures.

Brief Summary Text (26):

The hydrogel structures of this invention can be colored or dyed, can be made Radio opaque or complexed with iodine and other germicides and disinfectants.

Brief Summary Text (34):

The hydrogel structures of the present invention can also be colored, dyed, or rendered radio opaque and/or impregnated with disinfectant dyes and or complexed with iodine and/or other germicides.

Brief Summary Text (36):

Typical Radioopaque materials are those having high electron density and include, but are not limited to barium sulfate, bismuth suboxide, gold, and the like. Radioopaque materials are added in various amounts to the hydrogels, usually at the level of from about 1 to about 35 weight percent based on the total weight of the additive and the hydrogel.

Detailed Description Text (36):

Similar procedures as used in Examples 2 was used herein except that the polyvinyl alcohol solution was cast onto an assembly analogous to Band Aid.RTM. strips. Polyvinyl alcohol solution was poured onto the cotton gauze surface of the strips to create a hydrogel having a thickness of about 1 mm. The hydrogel was complexed with iodine by submersing the hydrogel into a solution of iodine. This article can be used in wound and burn healing applications providing the ability to dispense disinfectant in a controlled manner, provide protection for the injured skin, provide a non-adhering surface, and maintain moisture in a healing tissue.

Detailed Description Text (38):

The same procedure was used herein as was used in Example 17 except that the polyvinyl alcohol solution had a concentration of two weight %. A small amount of polyvinyl alcohol solution was poured onto an assembly analogous to the Band Aid strips of Example 17 and the excess polyvinyl alcohol solution was allowed to run off creating a coated or impregnated cotton gauze structure. The volume of the solution that was poured onto the cotton gauze surface of the strip was sufficient to completely coat the surface of the cotton fibers and create a thin hydrogel coating only on the fiber network without creating a continuous hydrogel surface or film supported by the structure. This approach allowed the complete surface modification of the cotton gauze and also allowed it to complex a sufficient amount of iodine by submersing the coated cotton gauze into a solution of iodine. This article can be used in wound and burn healing applications providing the ability to

dispense disinfectant in a controlled manner, provide protection for the injured skin, maintain moisture, provide a non-adhering surface to a healing tissue and allow air accessibility when necessary.

Detailed Description Text (40):

Similar procedures as was used in Example 6 were used herein except that the polyvinyl alcohol solution loaded with granular sugar was cast onto the assembly analogous to the Band Aid strips of Example 17. The polyvinyl alcohol solution was loaded with granular sugar and was placed onto the cotton gauze surface of the strip to create a cellular polyvinyl alcohol hydrogel on the surface of the cotton gauze having a height of about 2 mm. The hydrogel on the strip was obtained by cooling the assembly to about -15.degree. C. for 8 hours. Then, after extraction of the sugar, the hydrogel was complexed with iodine in the usual manner. This article can be used in wound and burn healing as well.

Current US Cross Reference Classification (1):

264/41



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Term:	L15 and gold and iodine		
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Display:	<input type="text" value="10"/>	Documents in	Display Format:	<input type="text" value="CIT"/>	Starting with Number	<input type="text" value="1"/>
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Search History

DATE: Friday, August 11, 2006 [Printable Copy](#) [Create Case](#)

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<u>L12</u>	leaching nanoparticles same membrane	0	<u>L12</u>
<u>L11</u>	11 and choloaurate	0	<u>L11</u>
<u>L10</u>	1 and choloaurate	0	<u>L10</u>
<u>L9</u>	11 and extracting gold	3	<u>L9</u>
<u>L8</u>	L6 and iodate	0	<u>L8</u>
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<u>L6</u>	1 and choroaurate	3	<u>L6</u>
<u>L5</u>	12 and choloaurate	0	<u>L5</u>
<u>L4</u>	2 and choloaurate	0	<u>L4</u>
<u>L3</u>	L2 and leaching	6	<u>L3</u>
<u>L2</u>	L1 and iodine	166	<u>L2</u>
<u>L1</u>	membrane and gold same particles and pores	1330	<u>L1</u>

END OF SEARCH HISTORY

US-PAT-NO: 4957943



DOCUMENT-IDENTIFIER: US 4957943 A

See image for Certificate of Correction

TITLE: **Particle**-filled microporous materials

DATE-ISSUED: September 18, 1990

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
McAllister; Jerome W.	Hudson	WI	N/A	N/A
Kinzer; Kevin E.	Cottage Grove	WI	N/A	N/A
Mrozinski; James S.	Oakdale	MN	N/A	N/A
Johnson; Eric J.	Woodbury	MN	N/A	N/A
Dyrud; James F.	New Richmond	WI	N/A	N/A

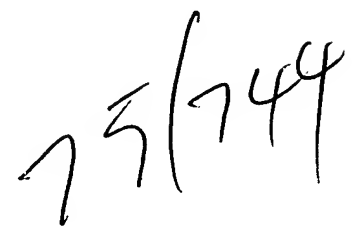
ASSIGNEE INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE
Minnesota Mining and Manufacturing Company	St. Paul	MN	N/A	N/A	02

APPL-NO: 07/258314

DATE FILED: October 14, 1988

INT-CL-ISSUED: [05] C08J009/28



INT-CL-CURRENT:

TYPE	IPC	DATE
CIPS	C08J3/20	20060101
CIPS	C08J3/205	20060101
CIPS	C08J9/00	20060101
CIPS	C08K3/00	20060101
CIPS	C08J9/28	20060101
CIPS	C08K3/08	20060101
CIPS	C08K3/22	20060101
CIPS	D01D5/247	20060101
CIPS	D01D5/00	20060101

US-CL-ISSUED: 521/64 , 521/61 , 521/82 , 521/91 , 521/92 , 428/315.5
, 428/315.7

US-CL-CURRENT: 521/64, 428/315.5 , 428/315.7 , 521/61 , 521/82 , 521/91
, 521/92

FIELD-OF-CLASSIFICATION-SEARCH: 521/61; 521/64 ; 521/92 ; 521/82 ; 521/91
; 428/315.5 ; 428/315.7

****See application file for complete search history****

REF-CITED:

U.S. PATENT DOCUMENTS

PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<u>2947646</u>	August 1960	Devaney et al.	106/1.19 N/A
N/A			
<u>3082109</u>	March 1963	Devaney et al.	106/1.19 N/A
N/A			
<u>3351495</u>	November 1967	Larsen et al.	136/146 N/A
N/A			
<u>3514607</u>	May 1970	Webster	250/108 N/A
N/A			
<u>3745142</u>	July 1973	Mahlman	260/41 N/A
N/A			
<u>3870593</u>	March 1975	Elton et al.	428/315.5 N/A
N/A			
<u>4247498</u>	January 1981	Castro	264/41 N/A
N/A			
<u>4324811</u>	August 1982	Lopatin et al.	428/220 N/A
N/A			
<u>4485838</u>	December 1984	Shoji et al.	164/462 N/A
N/A			
<u>4539256</u>	September 1985	Shipman	428/315.5 N/A
N/A			
<u>4550123</u>	October 1985	Lopatin et al.	521/64 N/A
N/A			
<u>4562108</u>	December 1985	Miyake et al.	428/212 N/A
N/A			
<u>4619963</u>	October 1986	Shoji et al.	524/439 N/A

N/A				
<u>4650730</u>	March 1987	Lundquist et al.	429/62	N/A
N/A				
<u>4726989</u>	February 1988	Mrozinski	428/315.5	N/A
N/A				

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
1100497	January 1968	GB	
1327602	August 1973	GB	

OTHER PUBLICATIONS

M. C. Williams & A. L. Fricki, Phase Separation Spinning of Polypropylene, SPE Journal , 28, Oct. 1972, p. 51.

D. Bennett, Nonwovens World, 2, Nov. 1987, p. 58.

Smolders, van Aartsen & Steenbergen, Kolloid-Z.u.Z. Polymere, 243, 14-20 (1971).

ART-UNIT: 153

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ABSTRACT:

A microporous particulate-filled thermoplastic polymeric article is provided. The article can be in the form of a film, a fiber, or a tube. The article has a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating pores. The microporous structure contains discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

34 Claims, 3 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 2

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Abstract Text - ABTX (1):

A microporous particulate-filled thermoplastic polymeric article is provided. The article can be in the form of a film, a fiber, or a tube. The article has a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating **pores**. The microporous structure contains discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

TITLE - TI (1):

Particle-filled microporous materials

Brief Summary Text - BSTX (2):

The present invention relates to **particle**-filled microporous materials such as films and fibers, a method for preparing same and articles prepared therefrom.

Brief Summary Text - BSTX (4):

It has been common practice to use fillers in polymers to produce a large variety of articles. Such articles contain a range of fillers such as, for example, highly stable and color-fast pigments, activated carbon sorbents, ion exchange resins, and fine silver **particles** for photographic films.

Brief Summary Text - BSTX (5):

U.S. Pat. No. 2,947,646 (Devaney et al.) discloses colloidal dispersions of metals in plastics which are prepared by depositing a thin coating of metal onto finely powdered plastic, working the metal-coated plastic powder into a plastic state, the working resulting in fragmentation of the metal coating into very small metallic **particles**, and fabricating into the final shape.

Brief Summary Text - BSTX (10):

U.S. Pat. No. 4,650,730 (Lundquist et al.) describes a battery separator having at least two plies, each in the form of a microporous sheet, at least one ply, formed from a polymeric composition comprising a polymer and, optionally, plasticizers, stabilizers, antioxidants, and the like but substantially no particulate filler and being capable of transforming to a substantially non-porous **membrane** at a temperature between about 80 and 150.degree. C., and at least one ply, formed from a polymeric composition

comprising a polymer and, optionally, plasticizers, stabilizers, antioxidants, and the like and preferably contains a large amount (greater than 20) weight percentage of solid, particulate filler.

Brief Summary Text - BSTX (11):

U.S. Pat. No. 3,745,142 (Mahlman et al.) describes a process for preparing highly filled polyolefins which comprises preparing a crystallizable olefin polymer having a specified **particle** size, adding to the olefin polymer about 50 to 1900%, based on the weight of olefin polymer of a solid, particulate, inorganic filler material which is insoluble in the olefin, which is solid at the melting point of the olefin polymer and which is in the form of **particles** of about 0.1 to 25 microns, shaping the resultant polymer-filler blend, and fusing the olefin polymer to form a continuous phase. The highly filled compositions can be prepared simply by adding the particulate inorganic filler directly into a dispersion, either aqueous or organic, of the olefin polymer and agitating to make the total dispersion uniform.

Brief Summary Text - BSTX (13):

U.S. Pat. Nos. 4,342,811 and No. 4,550,123 (Lopatin et al.) describe open-celled microporous sorbent-loaded textile fibers and films prepared by forming a melt blend, of the sorbent **particles**, the polymer and a selected diluent, e.g., in a batch-type blender such as a Sigma blade mixer or blending extruder such as a twin-screw compounding extruder, spinning or extruding and drawing down the fiber or film, and **extracting** the diluent.

Brief Summary Text - BSTX (14):

U.S. Pat. No. 4,562,108 (Miyake et al.) describes a heat-retaining moisture-transmissible water-resistant fabric having a fibrous substrate, a discontinuous polymer layer or a polymer layer having a multiplicity of interconnecting fine **pores**, and a polymer layer containing 15 to 70 weight percent, based on the weight of the polymer, of heat ray-reflecting fine metal pieces and having interconnecting fine **pores**. Alternatively, a specified microporous film layer may be interposed between the polymer layers. The metal-containing layer is prepared by solvent casting the polymer after simple mixing with an aluminum paste.

Brief Summary Text - BSTX (16):

Metal or metal oxide filled polymeric fabrics for X-ray absorption have been widely used to construct personal protective garments for a number of years. U.S. Pat. No. 3,514,607 describes composite shields against low energy X-rays which are sheets of a carrier material containing tin, antimony, **iodine**, barium, or a combination thereof and lead. The carrier material may be

flexible, e.g., a plastic or rubber material, or rigid, e.g., a plastic or a building material. The minimum content of carrier material needed to yield materials with acceptable mechanical strength is 16% by weight.

Brief Summary Text - BSTX (18):

U.S. Pat. No. 4,247,498 (Castro) discloses microporous polymers characterized by a relatively homogeneous, three-dimensional cellular structure having cells connected by pores of smaller dimension. The microporous polymers are prepared from thermoplastic polymers by heating a mixture of the polymer and a compatible liquid to form a homogeneous solution, cooling the solution under non-equilibrium thermodynamic conditions to initiate liquid-liquid phase separation, and continuing the cooling until the mixture achieves substantial handling strength.

Brief Summary Text - BSTX (19):

U.S. Pat. No. 4,539,256 (Shipman) discloses a microporous sheet material characterized by a multiplicity of spaced randomly dispersed, equiaxed, non-uniform shaped particles of the thermoplastic polymer, adjacent thermoplastic particles connected to each other by a plurality of fibrils of the thermoplastic polymer. The sheet materials are prepared by melt blending crystallizable thermoplastic polymer with a compound which is miscible with the thermoplastic polymer at the melting temperature of the polymer but phase separates on cooling at or below the crystallization temperature of the polymer, forming a shaped article of the melt blend, cooling the shaped article to a temperature at which the polymer crystallizes to cause phase separation to occur between the thermoplastic polymer and the compound.

Brief Summary Text - BSTX (22):

The present invention, in one aspect provides a microporous particulate-filled thermoplastic polymeric article which comprises a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

Brief Summary Text - BSTX (24):

The microporous particulate-filled articles may be provided as, for example, films, fibers, hollow fibers, and tubes. When the structure is in the form of a film, the film may be uniaxially or biaxially oriented. When the structure is in the form of a fiber, hollow fiber or tube, it may also be oriented. The articles of the invention have a network of interconnected passageways to provide communicating pores, with high effective pore size range, low fluid

flow resistance, broad pore size control and with up to 50 or more volume percent filler loading.

Brief Summary Text - BSTX (25):

The present invention, in a further aspect, provides a microporous particulate-filled thermoplastic polymeric filtration media comprising a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

Brief Summary Text - BSTX (26):

The present invention, in another aspect, provides a protective garment comprising a microporous particulate-filled thermoplastic polymeric fabric, the fabric comprising a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated. The microporous particulate-filled thermoplastic polymeric material may be bonded or laminated to a film, a woven, knitted, or nonwoven fabric or scrim to provide additional structural stability and durability.

Brief Summary Text - BSTX (27):

The present invention, in a further aspect, provides X-ray shielding material comprising a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized heavy metal X-ray absorbing particulate filler, the particulate filler being substantially non-agglomerated. Surprisingly, even when the densities of the X-ray shielding material of the invention are less than 0.5 percent of those of the heavy metal shielding, the materials of the invention are as efficient as a foil of the crystalline metal on a comparable weight per area basis.

Brief Summary Text - BSTX (28):

The present invention, in a further aspect, provides electromagnetic shielding material comprising a thermoplastic polymeric structure having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized heavy metal electromagnetic absorbing particulate filler, the particulate filler being substantially non-agglomerated. The particulate filler preferably absorbs electromagnetic waves having frequencies in the range of microwaves to X-rays.

Brief Summary Text - BSTX (38):

When liquid-liquid phase separation occurs, the cells comprise void spaces encased by fibrous, lacy, or semi-continuous boundaries with the filler **particles** attached thereto. Upon orientation, the cells become elongated in the direction of orientation. The cells of the oriented article are generally ellipsoidal in shape with an aspect ratio of major axis to minor axis greater than 1.0 and a major axis generally lying in a plane parallel to the surface of the article. The filled **particles** reside in and are attached to the thermoplastic polymer of the formed structure.

Brief Summary Text - BSTX (39):

When liquid-solid phase separation occurs, the material has an internal structure characterized by a multiplicity of spaced, randomly disposed, non-uniform shaped, equiaxed **particles** of thermoplastic polymer, adjacent **particles** throughout said material being separated from one another to provide the material with a network of interconnected micropores and being connected to each other by a plurality of fibrils consisting of the thermoplastic polymer. The fibrils elongate upon orientation providing greater spacing between the thermoplastic polymer **particles** and increased porosity. Again, the filled **particles** reside in and are attached to the thermoplastic polymer of the formed structure.

Detailed Description Text - DETX (8):

As used herein, "**particle**" refers to submicron or low micron-sized **particles**, also termed "particulate filler" herein, such **particles** having a major axis no larger than five microns.

Detailed Description Text - DETX (9):

As used herein, "discretely dispersed" or "colloidal suspension" means that the **particles** are arrayed substantially as individual **particles** with uniform spacings throughout a liquid or solid phase.

Detailed Description Text - DETX (10):

The submicron or low micron-sized **particles** useful in the present invention are capable of forming a colloidal dispersion with the compatible liquid and insoluble in the melt blend of the thermoplastic polymer and compatible liquid from which the articles of the invention are formed. The submicron or low micron-sized **particles** are preferably less than 5 microns in diameter, more preferably less than 3 microns in diameter, and most preferably less than about 1 micron in diameter. Useful **particles** include metals such as, for example, lead, platinum, tungsten, **gold**, bismuth, copper, and silver, metal oxides such

as, for example, lead oxide, iron oxide, chrome oxide, titania, silica and alumina, and blends thereof carbonaceous materials such as, for example, carbon black. Thermoplastic polymers useful in the present invention include olefinic, condensation and oxidation polymers Representative olefinic polymers include high and low density polyethylene, polypropylene, polyvinyl-containing polymers, butadiene-containing polymers, acrylate-containing polymers such as polymethyl methacrylate, and fluorine containing polymers such as polyvinylidene fluoride. Condensation polymers include polyesters such as polyethylene terephthalate and polybutylene terephthalate, polyamides such as nylon 6, nylon 11, nylon 13, and nylon 66, polycarbonates and polysulfones. Polyphenylene oxide is representative of the oxidation polymers which can be used. Blends of thermoplastic polymers may also be used.

Detailed Description Text - DETX (13):

The amount of filler **particles** in the thermoplastic polymer depends upon the amount of filler in the compatible liquid prior to melt blending and on the relative amount of thermoplastic polymer and compatible liquid in the blend. The amount does not depend upon liquid removal because the **particles** remain substantially entirely within the polymer structure. The amount of **particles** colloidally dispersed in the compatible liquid depends upon how well the **particles** are wet by the liquid, the surface area of the **particles**, and the proper choice of a dispersing aid or surfactant. Generally, for non-porous **particle**, a dispersion containing 40-50 volume percent **particles** can be achieved. The amount of filler in the polymer can be much greater than the amount of filler in the compatible liquid when the melt blend has a higher concentration of liquid than polymer.

Detailed Description Text - DETX (14):

The actual polymer concentration selected from within the predetermined concentration range for the liquid-polymer system being used is limited by functional considerations. The polymer concentration and molecular weight should be sufficient to provide the microporous structure which is formed on cooling with adequate strength for handling in further processing steps. The polymer concentration should be such that the viscosity of the liquid-polymer melt solution is suitable for the equipment used to shape the article. Generally, the polymer concentration in the compatible liquid is about 10 to 80 weight percent, which corresponds to a compatible liquid concentration of 20 to 90 weight percent. When high compatible liquid concentrations, i.e. 80 to 90 percent, are used in conjunction with high volume percent of filler in the compatible liquid, a very high, e.g., about 95 weight percent, concentration of the particulate filler in the thermoplastic polymer, relative to the liquid, can be achieved. For example, if the blend is 90:10 liquid/polymer by volume

and the liquid is 40 percent particulate filler by volume, then the resulting filled microporous article is, surprisingly, 80 percent particulate filler by volume after the liquid is removed. That the **particle**-filled microporous thermoplastic polymeric articles of the invention can contain such large amounts of particulate filler is unexpected because it is believed that **particle**-filled thermoplastic articles made by standard extrusion processes achieve only about 20 percent filler by volume.

Detailed Description Text - DETX (19):

In the microporous structures of the invention, the particulate filler is uniformly and discretely arrayed in the structure. For example, when the structure is spherulitic, **particles** are in both the spherulites and in the fibrils between them. Although the **particles** are firmly held in the polymeric structure, they are substantially exposed after liquid removal. In a structure, the distribution of **particles** is uniform wherever the polymer phase occurs. The **particles** substantially exist as individual, and not agglomerated, **particles** throughout the microporous structure. Agglomerates of 3 to 4 **particles** may occur, but their frequency is no more than in the compatible liquid dispersion prior to melt blending with the polymer. The average **particle** spacing depends upon the volume loading of the **particle** in the polymer.

Detailed Description Text - DETX (20):

The compatible liquid is removed from the material to yield a **particle**-filled, substantially liquid-free, microporous thermoplastic polymeric material. The compatible liquid may be removed by, for example, solvent extraction, volatilization, or any other convenient method, and the **particle** phase remains entrapped to a level of at least about 90 percent, more preferably 95 percent, most preferably 99 percent, in the microporous polymer structure.

Detailed Description Text - DETX (21):

The **particle**-filled microporous structures of this invention can be oriented, i.e., stretched beyond their elastic limit to introduce permanent set or elongation and to ensure that the micropores are permanently developed or formed. Orientation can be carried out either before or after removal of the compatible liquid. This orientation of the structures aids in controlling **pore** size and enhances the porosity and the mechanical properties of the material without changing the **particle** uniformity and degree of agglomeration in the polymer phase. Orientation causes the microporous structure to expand such that the porosity increases.

Detailed Description Text - DETX (22):

Orientation of films of the invention can be used as a process variable to control thickness and relatively thin microporous films can be produced. Thickness is particularly important for microporous film applications where selective fluid transport through the microporous film is desired, since the rate of transport is inversely proportional to the thickness. Decreasing thickness minimizes the hydrostatic resistance to flow through the film. Orientation enables production of thin films with minimal difficulty. Orientation also enhances the mechanical strength of the films which is beneficial in many microporous film applications. With increasing orientation, film thickness and resistance to flow are proportionately reduced, mechanical strength and porosity are proportionately increased, and the pore size range is extended with improved pore size control, so that an excellent balance of desired properties can be attained through selection of the amount of orientation to which the microporous film is subjected.

Detailed Description Text - DETX (23):

Particle-filled microporous films of the invention may be uniaxially or biaxially oriented. Preferably, the particle-filled microporous films are stretched at least about 10 percent, more preferably about 10 to 1000 percent. The actual amount of stretching required is dependent upon the particular composition of the article and the degree of porosity desired. Stretching of the structure is preferably uniform so that the oriented structure has uniform, controlled porosity. When the structures are uniaxially oriented, narrowing of the structure in the non-oriented direction generally occurs, such that stretching the film for example, a film, 50 percent does not result in a 50 percent increase in surface area, but something less than a 50 percent increase. Particle-filled microporous tubular film can be oriented, for example, using conventional film stretching devices as well as by inflating the tubular film during the extrusion process thereby reducing film thickness and expanding film dimensions radially. Particle-filled microporous fibers can be oriented, for example, by stretching the fibers in a lengthwise direction between a set of rolls with increasing speeds from 10 to 1000 percent before or after removal of the compatible liquid. The orientation is preferably dimensionally stabilized in the material using well-known techniques such as, for example, heating the material to a stabilizing temperature under restraint. The presence of filler particles has little measurable effect on the orientation process or on the mechanical properties of the resulting oriented article.

Detailed Description Text - DETX (24):

After removal of the compatible liquid and, optionally, orientation, the

resulting **particle**-filled microporous material may be modified by imbibition of various materials, such as, for example, liquids, solvent solutions, solvent dispersions, or solids. Such materials may be imbibed by any of a number of known methods which result in the deposition of such materials within the porous structure of the microporous material. The imbibed material may be physically entrapped within the microporous structure, chemically reacted with the polymeric material of the microporous structure, or attached to the particulate filler contained within the microporous structure. Examples of imbibing materials include medicaments, fragrances, antistatic agents, surfactants, and pesticides. The thermoplastic polymer may be imbibed with a urethane monomer which is then polymerized in place to provide a liquid-impermeable, vapor-permeable material.

Detailed Description Text - DETX (25):

The **particle**-filled microporous material of the invention may be further modified, either before or after removal of the compatible liquid, by depositing various materials on the surface thereof using known coating or deposition techniques. For example, the **particle**-filled microporous material may be coated with metal by vapor deposition or sputtering techniques or by materials such as adhesives, aqueous or solvent-based compositions, and dyes. Coating can be accomplished by such conventional coating techniques as, for example, roller coating, spray coating, dip coating, and the like.

Detailed Description Text - DETX (26):

Particle-filled microporous sheet materials of the invention may be laminated to various other materials such as, for example, woven, knitted, or non-woven fabrics, films, or to one or more additional layers of similar or other microporous sheet material to achieve, for example, desired thicknesses, porosity gradients, handling properties, and aesthetics. Lamination can be carried out using conventional techniques such as adhesive bonding, spot welding, or other techniques which do not undesirably interfere with the porosity or create undesirable porosity of perforations.

Detailed Description Text - DETX (27):

When **particle**-filled porous films of the present invention are prepared using a polyolefin as the thermoplastic polymer, a preferred method of producing multi-layered laminates results in a composite film that has high surface to surface bond strength of the laminates, and no interruptions of the porosity at the layer interfaces. The process used to form these multi-layered filled microporous polyolefin sheets takes place before stretching the polyolefin sheets and includes solvent exchanging the aforementioned compatible liquid used for the **particle** filled microporous film formation, for a volatile

solvent, then placing these wet sheets of polyolefin in intimate contact with each other, drying to remove the solvent and then stretching in one or more directions. Alternatively, the **particle** filled polyolefin sheets with the compatible liquid still in place, may be laid-up and placed in intimate contact with each other, the liquid removed by solvent extraction with drying of the solvent, and then stretched in one or more directions.

Detailed Description Text - DETX (28):

The **particle**-filled microporous material of the invention may be modified to produce a porous **membrane** having a gradient porosity therethrough, if the extruded film is rapidly cooled from one surface thereof immediately after extrusion, such as by bringing the surface into contact with a chilled casting wheel. The surface of the film in contact with, for example, the chilled casting wheel can be fused or sealed, while the opposite side remains porous. Orientation of this gradient porosity structure enhances the porosity differences from surface to surface. Films with such properties can be used, for example, for microfiltration or ultrafiltration or as protective films or tapes, having, for example, the porous side readily markable and the sealed side resistant to marking.

Detailed Description Text - DETX (29):

The **particle**-filled microporous materials of the invention are useful in a variety of applications where microporosity is desirable. For example, the microporous sheet materials can be used for ultrafiltration of colloidal matter, as filtering material for cleaning antibiotics, beer, oils, and bacteriological broths, and as diffusion barriers or separators in electrochemical cells. The microporous sheet material can also be used for sample collection in air analysis and for collection of microbiological specimens. When laminated to woven scrim material, the microporous materials can be useful for outerwear and for disposable protective garments for use, for example, in nuclear power plants, in hospitals, electronic clean rooms, or in areas where contact with hazardous chemicals or radiation can occur. The microporous sheet materials are also useful in surgical dressings, bandages, and other medical applications. In each of these applications the presence of a filler can enhance the performance of the material by adding either energy absorption or chemical reactivity.

Detailed Description Text - DETX (30):

The selection of the particulate filler is determined by the specific application for which the **particle**-filled microporous **membrane** is intended. The **particles** must be submicron or low-micron in size and are preferably chemically inert to the polymer and the compatible liquid. For example,

tungsten and lead oxide are efficient absorbers of hazardous radiation; aluminum and ferrite **particles** are useful for absorbing microwave energy and converting the microwave energy to heat; certain transition metal oxides, such as chrome oxide, are useful for converting solar energy to heat; and fine silver **particles** are useful as antibacterial agents.

Detailed Description Text - DETX (31):

The **particle**-filled microporous materials of this invention are useful as an extremely low-density shielding material for X-rays when the particulate filler is heavy metal-containing X-ray absorbing particulate. Even when the densities of the X-ray absorbent particulate-filled microporous porous films of this invention are less than 0.5 percent of those of the heavy metal shielding, the materials of the invention are as efficient as a foil of the crystalline metal on a comparable weight per area basis. Such low density composites of heavy metal X-ray absorbers is very desirable for production of practical protective fabric or garments.

Detailed Description Text - DETX (32):

The X-ray absorption efficiency of the materials of this invention appear to be as high as that of the pure metal at the same basis weight. This 100 percent equivalency holds for extremely thin layers, about 0.01 mm or less. This means that composites with the same absorption but less weight than currently available can be made. The metal-filled microporous **membranes** are vapor-permeable which makes garments much more comfortable for the wearer. The metal-filled porous **membranes** are soft, have a good hand, and are easily converted into garments even when the metal **particle** are present in high concentrations, e.g., 95 weight percent.

Detailed Description Text - DETX (33):

This surprising and unexpectedly high X-ray absorption efficiency of the **particle**-filled microporous articles of this invention is believed to be due to the discretely dispersed phase of colloidal-sized **particles** which are uniformly and discretely arrayed about the thermoplastic polymer phase. This degree of dispersion in the final article is a result of achieving a colloidal dispersion of the filler **particles** in the compatible liquid prior to melt-blending with the thermoplastic polymer.

Detailed Description Text - DETX (35):

The second, more difficult and more important, step of the process of this invention, is breaking down agglomerated **particles** to their primary **particle** size within the compatible liquid. This second step can be accomplished by milling, or grinding, the compatible liquid containing the particulate

material. Two types of mills useful for this milling are, for example, attritors and sand mills.

Detailed Description Text - DETX (36):

A surfactant is preferably added to the mixture of compatible liquid and particulate filler to aid in dispersing the particulate filler in the compatible liquid and in maintaining the particulate filler as discrete **particles**. Anionic, cationic or nonionic surfactants can be used. Preferably, the surfactant is a low molecular weight polymer which stabilizes the dispersion by steric interaction between the **particles** or a small ionic molecule which stabilizes the dispersion by a charge mechanism. Preferably, the surfactant is present in an amount of about 1 to 40, more preferably 2 to 20, weight percent based on the weight of the particulate filler. Useful surfactants include OLOA 1200, a polyisobutene succinimide, available from Chevron Chemical Co., Houston, Tex., Wayfos.TM. TD-100, available from Phillip A. Hunt Chemical Co., East Providence, R.I., and Kr-55, available from Kenrich Petrochemicals, Bayonne, N.J.

Detailed Description Text - DETX (37):

Milling reduces agglomerates to primary **particles** but does not break down large **particles** to smaller **particles**. Therefore, filtration of the milled dispersion may be necessary, if large **particles** are present.

Detailed Description Text - DETX (38):

When a liquid containing **particles** is pulled or pushed through a filter, a complex flow system is set up in which little streams of liquid move from the bulk phase into and through the **pores** of the filter and out the other side. Liquid flows more readily through the larger **pores**, so that these **pores** are more likely to participate in the filtration process. **Particles** suspended in the liquid are carried along in the flowing liquid by inertia. If the **particles** are small enough to pass through the holes of the filter, they pass out the other side and become part of the filtrate. Otherwise, the **particles** become impinged upon the surface of the filter, or become trapped within the interstices of the filter matrix.

Detailed Description Text - DETX (40):

In all of the examples to follow, the **particle**/compatible liquid milling was carried out at a relatively high viscosity where the grinding, or milling, process is much more effective. A typical dispersion contained about 20 percent by volume of **particles** in compatible liquid, and typical viscosity values were 10 poise at 0.1 sec.sup.-1 shear rate and 5 poise at 10 sec.sup.-1 shear rate, as measured using a Rheometrics Fluid Rheometer Model 7800 under

steady shear conditions. The grinding was carried out using an attritor or the sand mill.

Detailed Description Text - DETX (43):

Generally, in the attritor and in the sand mill, grinding times of 4 to 8 hours were required to achieve an adequate degree of dispersion, although, depending on the particulate used, grinding times of one hour or less are sufficient. The degree of dispersion was monitored by smearing a drop of the dispersion on a glass slide and viewing it with a laboratory microscope in a transmission mode at about 500X. At this magnification, micron **particles** can be resolved visually. The dispersion was deemed adequate when 95 percent or more of the **particles** in the dispersion existed as primary **particles**, rather than agglomerates.

Detailed Description Text - DETX (44):

A surfactant was utilized during the dispersion step to reduce viscosity at the higher volume loadings and to stabilize the dispersion toward flocculation. The surfactant utilized in most of the examples that follow was OLOA 1200, a polyisobutene succinimide, available from Chevron Chemical Co., Houston, Tex. Surfactant levels were about 10 percent by volume of the **particles**. In addition to being an effective dispersing aid for a variety of **particle**/organic liquid combinations, it is thermally stable at the melt blend temperatures utilized herein.

Detailed Description Text - DETX (45):

In the examples that follow, it was necessary to filter the dispersions of **particles** in the compatible liquid to remove any large **particles**. A Model C3B4U 3 micron rope-wound filter made by Brunswick Technetics in Timonium, Md., was used just prior to melt-blending the dispersion with the thermoplastic polymer. The filtration step also removed hard agglomerates that had not been reduced by the milling to diameters less than 3 microns. This resulted in a more uniform finished article and allowed the dispersions to be metered under pressure by close tolerance gear pumps during the extrusion process without frequent breakdowns due to large **particles** clogging the pump. In all cases, less than 10 percent by weight of the dispersed **particles** was removed by this filtration step. After filtering, the concentration of **particles** in the compatible liquid was determined by measuring density with a Model DMA-4S Mettler/Paar density meter manufactured by Mettler Instrument Co. Hightsdown, N.J.

Detailed Description Text - DETX (59):

In this technique, three scans (heat, cool, heat) are run on the material of

interest and a control material produced under similar conditions but without the filler **particles**. The heat of fusion is determined for each of the scans and filler concentration calculated using the following equation:
$$\frac{X_{\text{sub}}(\text{filler})}{\Delta H_{\text{sub}}(\text{control})} = \frac{\text{wt-\% filler in the sample}}{\text{heat of fusion of the control sample (cal/g)}}$$

Detailed Description Text - DETX (64):

The microstructure of the **particle** filled microporous materials was examined using an ISI model Super-III A scanning electron microscope (SEM). The materials were prepared for SEM analysis by freeze fracturing under liquid nitrogen, mounting on SEM stubs, and vapor coating with approximately 150 to 200 angstroms of pure **gold**. The size and spacial distribution of the metal and metal oxide **particles** were determined by using back-scattered electron imaging with the SEM on uncoated materials. The resulting image is bright spots (resulting from the metal and/or metal oxide **particles**) on a dark background.

Detailed Description Text - DETX (66):

A mixture of 499 g of submicron tungsten powder, having an average **particle** size of about 0.5 micron, obtained from Union Carbide Corp., Danbury, CT, 402 g white mineral oil having a density of 0.87 g/cc, and 20 g Wayfos.TM. TD-100 surfactant, available from Phillip A. Hunt Chemical Corp., East Providence, R.I., was prepared. A dispersion of the tungsten powder in the mineral oil was achieved by milling with the Igarashi attritor for 6 hours, at 2000 RPM. Light microscopy at 300X showed a dispersion comprised of primary **particles** having a diameter of less than about 1 micron. This dispersion was diluted with additional mineral oil to a concentration of 5 weight percent tungsten and filtered using a 3 micron filter. Density measurements before and after filtering showed no significant loss of tungsten. The density of the dispersion was 0.93 g/cc.

Detailed Description Text - DETX (68):

The film was restrained in a frame and washed with 1,1,1-trichloroethane to remove the mineral oil. The effluent was clear indicating that substantially all of the tungsten **particles** remained with the polyethylene. The restrained, washed film was dried to remove any residual 1,1,1-trichloroethane. The dried film was oriented by stretching at a temperature of 93.degree. C., a preheat time of about 1 minute, a stretch rate of 30 cm/min, and a stretch ratio of 3:1 in the machine direction and 3:1 in the transverse direction. While maintained at this stretch ratio, the oriented film was then heat set at a temperature of 100.degree. C. The resulting tungsten-filled microporous polyethylene film was evaluated for tungsten concentration using a differential scanning calorimeter and for thickness and porosity. The results are set forth in Table 1.

Detailed Description Text - DETX (69):

Scanning Electron Microscopy, SEM, analysis at a magnification of 5000X of the porous filled polyethylene film showed a spherulitic structure indicative of liquid-solid phase separation, with spherulite diameters of about 2 microns spaced 1 to 5 microns apart. Back scattered SEM analysis at a magnification of 2000X showed that the tungsten particles were discretely dispersed in the polymer, with most particles smaller than 1 micron and none larger than 3 microns.

Detailed Description Text - DETX (71):

Control experiments showed that the X-ray absorption contributions from the polyimide film in the foil sample and from the polyethylene in the 8 layers of porous film, were less than one percent. These results show that a small amount of sub-micron tungsten particles, dispersed throughout a volume 2000 times larger than an equivalent volume of crystalline tungsten foil, has an absorption efficiency equivalent to that of the foil. This, in turn, shows that the tungsten-filled microporous film of this example has an extremely uniform dispersed phase of sub-micron particles on a scale of the primary particle size, i.e., 1.0 micron.

Detailed Description Text - DETX (79):

A PbO content of 93 weight percent is a filler content of 57 volume percent, a remarkably high loading. The limiting factor on filler levels in many processes is the loss of mechanical properties at about the 20 volume percent level. This is believed to be due to the filler particles not being sufficiently dispersed in the polymer. Although good dispersion in plasticizers are easily achieved, high plasticizer contents also tend to deteriorate mechanical properties. The strength of the process of this invention is that large volumes of filler can be introduced effectively with relatively large volumes of plasticizer, i.e., the compatible liquid, without loss of mechanical properties because the plasticizer is not present in the final article. Further, this removal is accomplished without substantial removal of the filler from the porous thermoplastic matrix.

Detailed Description Text - DETX (82):

In Example 4, a dispersion of tungsten particles, 0.8 to 1.0 micron diameter of Example 3, was prepared using 7.25 Kg of tungsten, 254 g OLOA surfactant, and 4.79 Kg mineral oil using the 0.5 gallon vertical sand mill operating at 3000 RPM for 7 hours. This mixture was diluted with mineral oil to a final density of 1.75 g/cc. Filled porous high density polyethylene film was extruded and cast as described in Example 1 using a melt blend volume ratio of

33:67 polymer to dispersion. After casting and cooling to effect liquid-solid phase separation and film solidification, the film was fed continuously into an extractor tank containing 1,1,1-trichloroethane to remove the mineral oil. The extraction tank was plumbed such that fresh 1,1,1-trichloroethane was continuously added and the 1,1,1-trichloroethane which was saturated with mineral oil was removed counter-current to the film. The next step in the continuous process was drying with forced air at a temperature of 88.degree. C. Thereafter, the filled porous film was stretched first in the machine direction and then in transverse direction, both at a stretch ratio of 3:1. Machine direction stretching was at ambient temperature and the transverse direction stretching at a range of 79 to 101.degree. C. Characterization data is given in Table 5.

Detailed Description Text - DETX (87):

An Ultra-Turrax Model SD-45 high speed shear mixer from Tekmar Co., Cincinnati, Ohio, was used to mix mineral oil containing 5 weight percent of 0.5 micron copper **particles** from Alfa Products, Danvers, Mass , for 1 hour at 100 percent power. Porous high density polyethylene film was prepared as in Example 1, with a melt blend ratio of 18.1:81.9 polymer to dispersion. The cast film was washed with 1,1,1-trichloroethane to remove the mineral oil and biaxially oriented as described in Example 1. Characterization data is given in Table 7.

Detailed Description Text - DETX (88):

Backscatter SEM was used to characterize the film at both 500X and 2000X. The results showed that most of the copper **particles** were in agglomerates of 5 to 10 microns in diameter and that the agglomerate distribution was non-uniform.

Detailed Description Text - DETX (89):

This data can be compared to the backscatter SEM data of the filled porous film of Example 1 wherein the film was made in the same manner as this example except the submicron **particles** were tungsten and were dispersively milled into the mineral oil. The 500X and 2000X SEM data of Example 1 shows that most of the tungsten occurs as primary **particles** about 1 micron in diameter or smaller and that these are uniformly dispersed within the polymer. There were a few agglomerates of tungsten containing 3 or 4 primary **particles** but these were no larger than 3 microns in diameter. Photomicrographs of the SEM data at 2000X are shown in FIG. 2 for the copper of this example and in FIG. 3 for the tungsten of Example 1. The streaks in both photographs are artifacts produced by shadowing from the polyethylene matrix.

Detailed Description Text - DETX (91):

A mixture of 150 g tungsten powder (particle diameter 0.8 to 1.0 microns, available from Teledyne Wah Chang, Huntsville, Ala.), 360 g dibutyl phthalate, and 5.0 g OLOA surfactant was prepared. The dispersion was milled using the Igarashi attritor at 1800 rpm for 6 hours. A blend of 394 g polyvinylidene fluoride (Soltex 1011, available from Solvey, Inc.), and 394 g of the tungsten powder/dibutyl phthalate dispersion were batch mixed for 4 hours at 200.degree. C. under a nitrogen atmosphere. The blend formed a homogeneous mixture which was pressed between plates heated to 200.degree. C. to form a film approximately 0.6 mm thick which was quenched in a 5.degree. C. water bath to initiate thermodynamic non-equilibrium solid-liquid phase separation and solidification.

Detailed Description Text - DETX (94):

A mixture of 150 g tungsten powder, (particle diameter 0.8 to 1.0 microns, obtained from Teledyne Wah Chang, Huntsville, Ala.), 360 g tallow amine (Armostat 310, available from Armat Chemical Co., Chicago, Ill.), and 5.2 g OLOA surfactant was prepared. The dispersion was milled using the Igarashi attritor at 1800 rpm for 6 hours. An additional 125 g of tallow amine was added to 402 g of the tungsten/tallow amine dispersion. A blend of 173 g polypropylene (Profax 6723, available from Himont, Inc.), and 517 g of diluted dispersion were batch mixed for 4 hours at 200.degree. C. under a nitrogen atmosphere. The blend formed a homogeneous mixture which was pressed between plates heated to 180.degree. C. to form a film approximately 0.6 mm thick which was quenched in a 5.degree. C. water bath to initiate thermodynamic non-equilibrium liquid-liquid phase separation and solidification.

Detailed Description Text - DETX (97):

A mixture of 1350 g tungsten powder (particle size 0.8 to 1 micron, available from Teledyne Wah Chang, Huntsville, Ala.), 350 g mineral oil having a density of 0.87 g/cc, and 44 g OLOA 1200 surfactant was prepared. A dispersion of the tungsten powder in the mineral oil was achieved using the Igarashi attritor at 1800 rpm for 6 hours. 100 g of the dispersion was diluted with an additional 323 g of mineral oil. A blend of 138 g polypropylene (Profax.sup.TM. 6723, available from Himont, Inc.), 0.35 g nucleating agent (Millad.TM. 3905, available from Milliken Chemical Co.) and 414 g of diluted dispersion were batch mixed for 4 hours at 200.degree. C. under a nitrogen atmosphere. The blend formed a homogeneous mixture which was pressed between plates heated to 180.degree. C. to form a film about 0.6 mm thick which was quenched in a 5.degree. C. water bath.

Detailed Description Text - DETX (100):

A mixture of 600 g tungsten powder (particle size 0.8 to 1 micron, available from Teledyne Wah Chang, Huntsville, Ala.), 300 g triethylene glycol, and 10.5 g OLOA 1200 surfactant was prepared. A dispersion of the tungsten powder in the triethylene glycol was achieved using the Igarashi attriter at 1800 rpm for 6 hours. A blend of 170.5 g nylon 6 (Nycoa.TM. 589, available from Nylon Corporation of America, Manchester, N.H.), and 500 g of dispersion were batch mixed for 4 hours at 220.degree. C. under a nitrogen atmosphere. The blend formed a homogeneous mixture which was pressed between plates heated to 220.degree. C. to form a film about 1.2 mm thick which was quenched in a 5.degree. C. water bath.

Claims Text - CLTX (11):

5. A microporous particulate-filled thermoplastic polymeric article which comprises a thermoplastic polymeric structure made by the method of claim 1 having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

Claims Text - CLTX (17):

11. The article of claim 5 wherein said structure comprises a multiplicity of spaced, randomly disposed, non-uniform shaped, equiaxed particles of thermoplastic polymer, adjacent particles throughout said structure being separated from one another to provide the structure with a network of interconnected micropores and being connected to each other by a plurality of fibrils consisting of said thermoplastic polymer.

Claims Text - CLTX (27):

21. The article of claim 20 wherein said metal is lead, platinum, tungsten, gold, bismuth, copper or silver.

Claims Text - CLTX (37):

31. A microporous particulate-filled thermoplastic polymeric filtration media comprising a thermoplastic polymeric structure made by the method of claim 1 having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

Claims Text - CLTX (38):

32. A protective garment comprising a microporous particulate-filled thermoplastic polymeric fabric, the fabric comprising a thermoplastic polymeric

structure made by the method of claim 1 having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized particulate filler, the particulate filler being substantially non-agglomerated.

Claims Text - CLTX (39):

33. X-ray shielding material comprising a thermoplastic polymeric structure made by the method of claim 1 having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized heavy metal-containing X-ray absorbing particulate filler, the particulate filler being substantially non-agglomerated.

Claims Text - CLTX (40):

34. Electromagnetic shielding material comprising a thermoplastic polymeric structure made by the method of claim 1 having a plurality of interconnected passageways to provide a network of communicating pores, the microporous structure containing discrete submicron or low micron-sized heavy metal electromagnetic absorbing particulate filler, the particulate filler being substantially non-agglomerated.